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(71) Applicant
China Steel Corporation

(Incorporated in Taiwan)

No 1 Chung-Kang Road, Hsiao-Kang District, Kaohsiung City, Taiwan, Province of China

(72) Inventor
Hong-Bin Chen

(74) Agent and/or Address for Service
D Young & Co
10 Staple Inn, London, WC1V 7RD, United Kingdom

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(54) **Determination of metal properties during phase transformation**

(57) An apparatus 100 for measuring the internal friction variation with temperature induced phase transformation comprises a device 23 for fixedly mounting one end of a metal specimen 31 on the base 21 of a vacuum housing 22 and the other end on a mounting device 30 of a torsion pendulum device 27, 28, 29. The pendulum device is suspended from a counterbalanced lever 25 which is provided with core 47 movable in solenoid 46 to measure thermal expansion. A triggering device 42 controlled by a programmed controller 32 initiates and preferably maintains natural frequency vibration of the alloy specimen 31. An internal friction measuring device 43 provided with a motion detector 44 measures internal friction and sends the data to a recorder 41. A heating device 33 controlled by programmed controller 32 heats specimen by a resistance or induction heater 34. A cooling gas e.g. liquid nitrogen is sprayed on to the specimen under the control of programme controller 32. A thermo-couple 40 embedded in the fixed end of the specimen supplies a signal to controller 32 and the recording device 41. Magnetic variations of the specimen with temperature are detected by a detecting device 48 having induction coils 49 around the specimen. The apparatus enables internal friction, thermal expansion, and magnetic properties of the specimen to be continuously measured as the specimen undergoes a predetermined temperature regime under the control of the programmable controller 32.

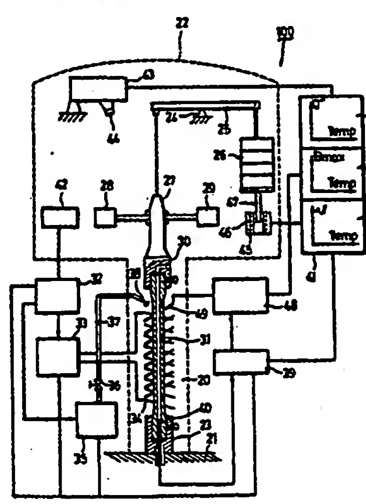

FIG. 2

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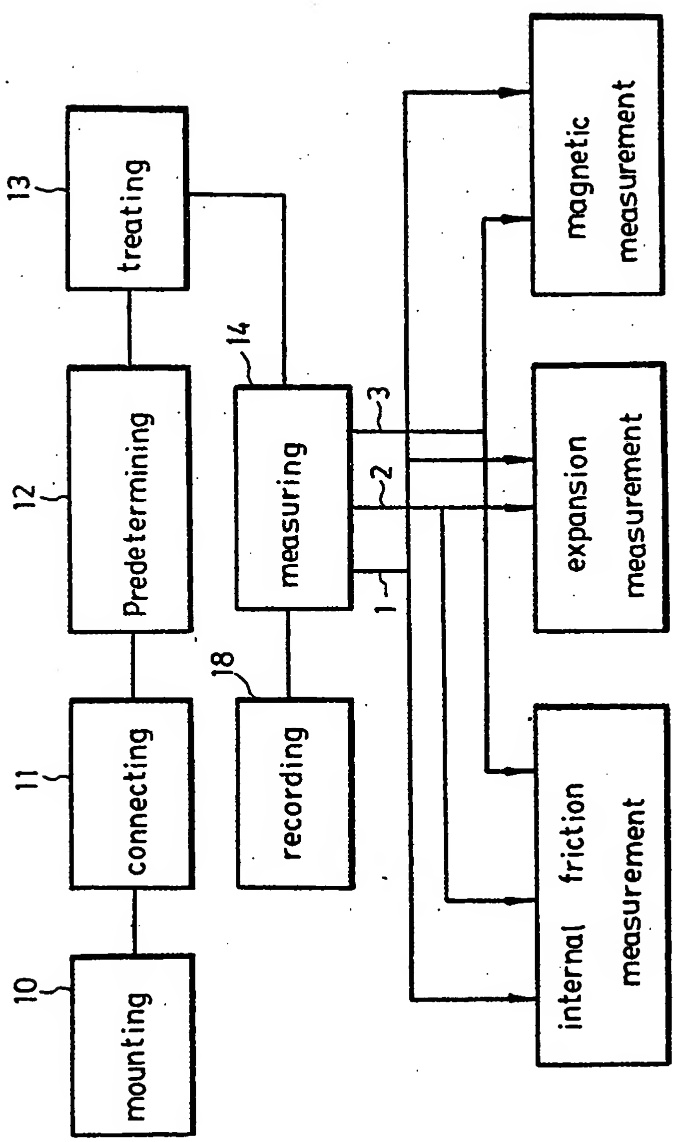

10
mounting
11
connecting
12
Predetermining
13
treating
14
measuring
18
recording
1
2
3
magnetic measurement
expansion measurement
internal friction measurement

FIG. 1

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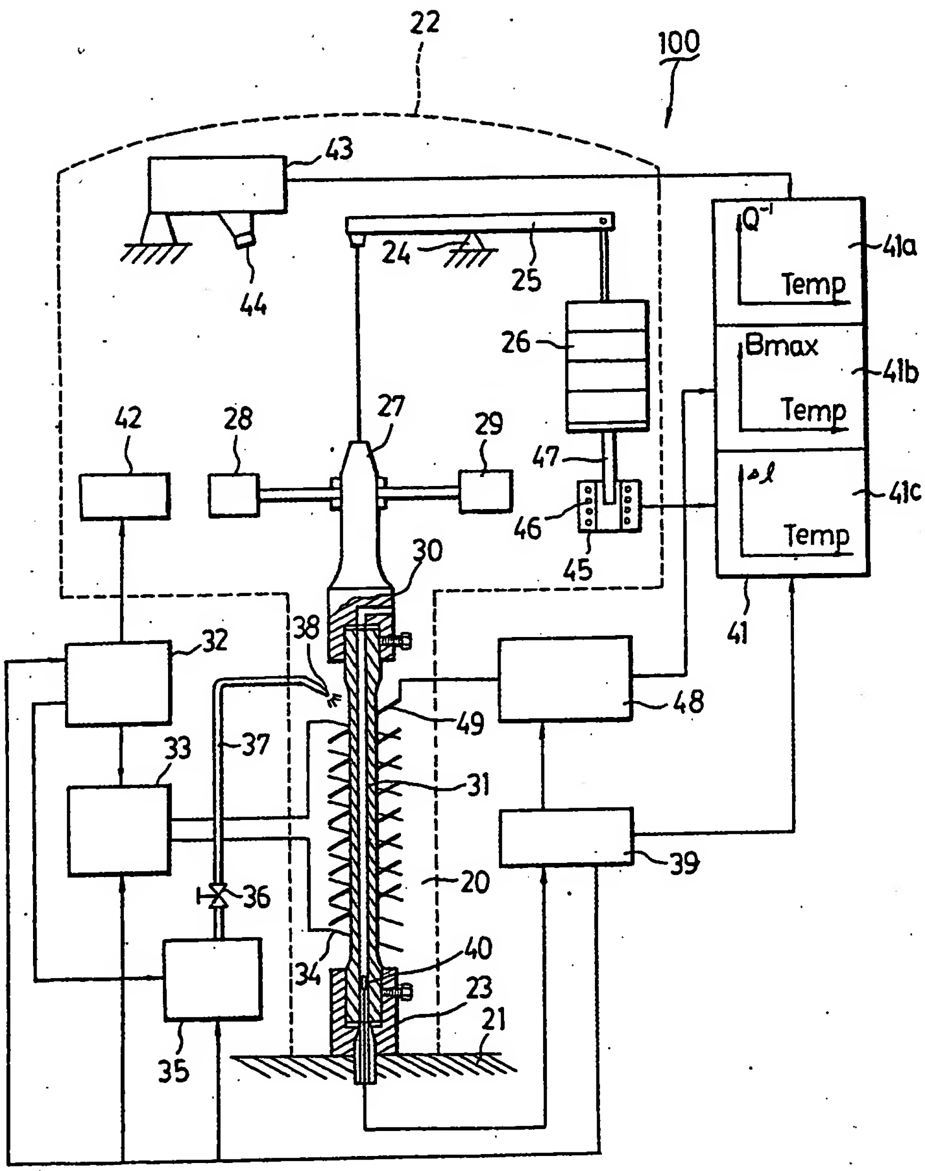

FIG . 2

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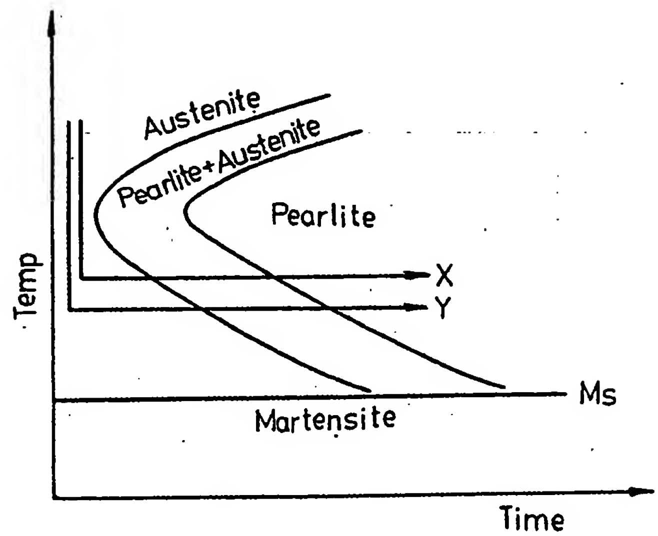

FIG. 3

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TITLE: DETERMINATION OF METAL PROPERTIES DURING PHASE TRANSFORMATION

This invention relates to a method for determining the properties of metals during its phase-transformations, and more specifically to a method by which the internal friction behavior, thermal expansion curve and/or the magnetic property variation curve of an alloy can be measured at high temperatures where metal alloy transformation occurs. This invention further relates to an apparatus which conducts the steps of the above-mentioned method and measures the internal friction behavior of an alloy during its phase transformation.

The measuring techniques of internal friction that has been developed in the past few decades, i.e. the torsion pendulum method, the transverse vibration method and others for measuring internal friction behavior of metals, both provide significant contributions to the study of metals. The techniques of measuring internal friction are used to measure the situations of solid solutions in a metal alloy, such as the solute content of nitrogen and/or of carbon in steel, the distribution and diffusion of some elements in an alloy, and other defects in a metal, etc.

Therefore, due to the fact that those new techniques provide meaningful measured results of the inside situation of metals, persons in the art can now understand metals much better than before.

Although internal friction measuring has been widely applied in the study of metals, the measuring condition needs to be improved in at least two aspects. Firstly, during a heat treatment, such as quenching, tempering, and annealing, metals are treated at several different temperatures which cause the structural change, but the internal friction measuring of the metal specimens has only been conducted after the whole heat treatment procedure has been completed. That is to say, for example, the known skills can't measure the internal friction behavior of the incubation period of a over cooled austenite transformations during the heat treatment, but only provides the measured data of heat treated specimens whose phase is in a steady and/or final state. Secondly, due to the limitation of experimental equipment, the known skills can not be performed at high temperatures, such as 750°C to 1100°C. At these high temperatures the known equipment can not measure the in-situ internal friction behavior of a metal specimen during the heat treatment procedure.

According to the present invention, a novel internal friction measuring method comprises the following

steps: 1) treating a metal specimen on a pre-determined heat-treatment schedule including a high temperature range; and 2) measuring the internal friction behavior of the metal specimen so as to obtain different internal friction values during the phase transformation of the specimen. Furthermore, according to the present invention, an apparatus for measuring the internal friction behavior of a metal specimen during the metal phase transformations comprises: a specimen mounting means having a vacumn space thereon for allocating a metal specimen; a programmable controlling means in which measuring modules and temperature changing programs are provided for controlling pre-determined operations; means for triggering the metal specimen and then measuring the internal friction behavior of the specimen; a temperature controlling means controlled by the programmable controlling means for controlling the temperature variations applied to the metal specimen; and means for recording the measured internal friction values corresponding to different temperatures, whereby the in-situ internal friction data of the metal specimen during its phase transformation can be obtained.

Moreover, the method of this invention can also include steps of measuring the expansion curve as well as the magnetic curve of the metal specimen. The

apparatus of this invention can further include means for measuring the expansion curve of the specimen and means for measuring the magnetic property variation curve of the specimen.

With the present invention the above-stated difficulties of the known skills can be overcome.

Accordingly, the primary object of the present invention is to provide a method for measuring the internal friction behavior of a metal alloy during the period of alloy phase transformation.

Another object of the present invention is to provide a method for measuring both the internal friction behavior, and the expansion and/or magnetic property variation curve of a metal alloy during a heat treatment procceding so as to confirm whether the measurment is taken in the incubation period or other period of phase transformation of the metal.

Still another object of the present invention is to provide a method for measuring the in-situ internal friction behavior of a metal alloy during a heat treatment proceeding.

Yet another object of the present invention is to provide an apparatus which conducts the measurements of the in-situ internal friction, expansion curve, and magnetic property variation curve of a metal alloy during heat treatment such that internal friction variations, from one phase to another, of the alloy can be clearly measured. In addition, the present

apparatus can also perform the same task as the conventional one, that is measuring the internal friction of the heat treated metal alloy whose phase is in a steady state.

These and other objects, features and characteristics of the present invention will become more obvious from the following detailed description of a preferred embodiment of the invention in conjunction with the drawings.

Fig. 1 is a flow-chart of block diagrams for illustrating the method of this invention;

Fig. 2 is a scheme for illustrating an embodiment of the apparatus of this invention; and

Fig. 3 is a chart of a time-temperature-transformation curve for illustrating the phase transformations of an iron-based alloy, in which lines X and Y are predetermined conditions, using the method of this invention to investigate the phase transformation for different periods with an isothermal in-situ measuremental procedure.

In Fig. 1 a flow chart is shown of the method according to this invention in which numeral 10 indicates a step of mounting a metal specimen on a

measuring device; numeral 11 indicates a step of connecting a temperature measuring device, such as a thermal couple, to the metal specimen; numeral 12 indicates a setp of predetermining a temperature variation pattern of the specimen by a programmable temperature controller; numeral 13 indicates a step of treating the metal specimen under the control of the temperature controller; numeral 14 indicates a step of measuring the in-situ internal friction behavior, expansion curve and/or magnetic property variation curve of the metal specimen; and numeral 18 indicates a step of recording the measured data so as to provide a better understanding of metals. In the heating step 12, the metal specimen may be heated by several different treatments such as annealing, tempering or quenching. No matter what kind of heat treatment is used, the metal specimen is kept at its original position so that an in-situ measurement can be conducted. In the connecting step 11, the temperature measuring device connected to the metal specimen must not cause any important unexpected external friction to the metal specimen. This is in contrast to the torsion pendulum internal testing apparatus, in which the part of the specimen on which the thermal couple is connected will not experience any torsion, i.e. the place near the torsion center of the metal specimen and the place near the fixed end of the specimen. As a matter of fact, this connection between the thermal couple and the

specimen is also one of the characteristics of the invention. The thermal couple, according to the invention, is preferably embeded into the fixed end of the specimen so as to get an accurate measurement; particularly during quenching proceedings. In the measuring step 14, three different combinations of measurements can be optioned, namely, the measurements of the internal friction, the expansion curve and the magnetic property variation curve indicated by numeral 1; measurements of the internal friction and the expansion curve indicated by numeral 2; and measurements of the internal friction and the magnetic property variation curve indicated by numeral 3.

Turning to Fig. 2, there is shown a schematic diagram of an apparatus 100 which is a preferred embodiment of this invention. The apparatus 100 comprises a torsion internal friction testing device 20 in which a vacuum housing 22 is mounted on a supporting base 21 so that an internal friction measurement can be conducted under the vacuum condition. In the housing 22, a specimen mounting device 23 is provided on the supporting base 21. At the upper portion of the torsion internal friction testing device 20 there is provided a pointed support 24 on which a lever 25 is pivoted. A counter balance 26 is connected with one end of the lever 25 and a torsion pendulum device 27 is suspended from the other end of the lever 25. A pair of arms

28,29 are respectively extended from the torsion pendulum device 27 in opposite directions. At the lower portion of the torsion pendulum device 27 there is provided a second specimen mounting device 30 so that a bar-shaped metal specimen 31 can be mounted between the two mounting devices 23 and 30.

The apparatus 100 further comprises a programmable controlling device 32 by which a desirable pattern of measurement can be pre-determined. In other words, with the programmable controlling device 32, a user can option any one of the combinations of the measurements as described above in Fig.1. Furthermore, the programmable controlling device 32 can also pre-determine the conditions of heating, cooling, and isothermal heating in the heat treatment of the metal specimen 31 so as to control the specimen 31 at a desired temperature during the whole measuring proceeding. A heating device 33 is controlled by the programmable controlling device 32 to heat the specimen 31 and can either be an electric heating wire 34 or a induction coil. A cooling device controlled by the programmable controlling device 32 includes a controlling valve 36, a conduit 37 and a nozzle 38. In the cooling operation, using the control valve 36, a cooling gas, such as liquid nitrogen, will be sprayed out of the nozzle 38 onto the heated specimen. A temperature measuring device 39 is connected with a thermal couple 40 whose one end is passed through the

mounting device 23 and then embeded into the torsion center of the specimen 31 as clearly shown in Fig. 2. In this way, this connection between the thermal couple 40 and the specimen 31 will not cause some unexpected external friction to the metal specimen. The measured data of the temperature measuring device 39 will be provided not only to the programmable controlling device 32 but also to a recording device 41.

Preferably, the measurement conducted by the torsion internal friction testing device 20 is proceeded under 10^{-2} to 10^{-3} Torr of vacuum.

A triggering device 42 is provided in the torsion internal friction testing device 20 and controlled by the programmable controlling device 32. The triggering device 42 will cause a torsion to the arms 28,29 of the torsion pendulum device 27 until it is equal to the natural frequency of the torsion pendulum of the metal specimen 31 so as to obtain the desired torsional vibration of the metal specimen 31. In addition, an internal friction measuring device 43 provided with a motion detecting device 44 measures the internal friction values of the metal specimen 31 and sends the measured data to the recording device 41. Therefore, after a heat treatment is applied to the metal specimen 31, a curve 41a showing the internal friction curve of the specimen can be obtained.

Since the damping of the triggered specimen 31 may

be fast, some difficulties may be created in proceeding with the measurement. Thus, in accordance with the present invention, it is preferred that when the designed torsional vibration of the triggered specimen 31 is reached, an external energy be applied to make the triggered specimen keep its constant amplitude torsional vibration. The value of the applied external energy should be the same as the value of the internal friction of the specimen. This novel method of the present invention will render the internal friction measurement more accurate.

In addition, a dilatometer 45 includes a solenoid coil 46 connected with the recording device 41, and a core 47 suspended from the underside of the counter balance 26 and extended into the solenoid coil 46. Following the variation of temperature during the heat treatment proceeding, the expansion of the specimen 31 will change, making the lever 25 move clockwise or counterclockwise, and, making the core 47 move downward or upward. The dilatometer 45 will note the changes in magnetic conductivity which represent corresponding up-down movement of the core 47 and will send the resulting measured data to the recording device 41. Therefore, a curve 41c showing the relation between the variation of temperature and the expansion of the metal specimen will be obtained by the recording device 41.

The apparatus 100 according to this invention further comprises a magnetic detecting device 48 having

induction coils 49 surrounding the metal specimen 31. Following the variation of temperature, the induction coils 49 will induce changes in the magnetic flux of specimen 31 and will send the data concerning these changes to the recording device 41. Therefore, a curve 41b showing the relation between the variation of temperature and the magnetic property of the specimen 31 can be obtained.

From the above described apparatus, it should be appreciated that due to the application of the programmable controlling device 32, one can easily predetermine the items and patterns of the measurement and the variation of temperature. According to this invention, the apparatus 100 is capable of not only measuring the internal friction behavior, expansion and/or the magnetic property change of the metal specimen during the heating, cooling and isothermal heating proceedings of the heat treatment, but also capable of keeping the specimen 31 in-situ while conducting those measurements. Thus, the apparatus provides a lot of valuable measured data which can not be obtained with known skills and is significantly meaningful to the study of metals.

As an example, Fig. 3 shows a time-temperature-transformation curve of an iron-based alloy during heat treatment which illustrates the phase transformations of the alloy. By using the traditional method, a

skilled person may obtain the results of the phase transformations shown in Fig. 3. Many real situations during which can not be understood. For instance, when the crystal structure of the iron-based alloy transforms from the austenite phase into the pearlite phase, persons skilled in the art find it difficult to get information on transformation process, especially,in such period as the incubation period.

It is clearly shown that in Fig. 3, lines X and Y represent repectively, a specimen of an iron-based alloy cooled rapidly from a higher temperature to a lower temperature and then kept in an isothermal proceeding for a certain period. By using the skills of this invention disclosed previously, the detailed measurements of the internal friction of the iron-based alloy can be obtained not only in the situation of a single phase but also in process of phase transformation, such as the austenite range to the range of austenite and perlite, and then eventually to perlite range. In other words, according to this invention, after changes of the magnetic flux and/or expansion curve are measured, one will know that the phase transformation has been effected. Therefore, the obtained values of the internal friction of the alloy before, during, and after the phase transformations will be significantly useful for the further study of metal.

It should be understood that any person skilled in

the art may make some minor modifications in light of the previous description of this invention. However, such modifications shall fall into the scope of the appended claims.

CLAIMS

1. A method for measuring the internal friction behavior of a metal specimen during the phase transformation of the metal specimen comprising the following steps:

(a) treating the metal specimen in a heat treatment whose time-temperature patterns are predetermined by a programmable controlling device; and

(b) measuring the in-situ internal friction of said metal specimen as it corresponds to the variation of temperature; whereby the internal friction behavior, particularly during the phase transformation of the metal specimen, can be obtained.

2. A method as claimed in Claim 1 wherein the treating temperature of said step (a) can be up to 950° C.

3. A method as claimed in Claim 1 wherein said patterns of predetermined time-temperature include the settings of heating, cooling, and isothermal heating.

4. A method as claimed in Claim 1 wherein the way of measuring mentioned in said step (b) is preferably the inverse torsion pendulum internal friction testing method.

5. A method as claimed in Claim 1 further comprising a step (c) of measuring the in-situ thermal expansion of said metal specimen.

6. A method as claimed in Claim 1 further comprising a step (d) of measuring the in-situ magnetic property of

said metal specimen.

7. A method as claimed in Claim 5 further comprising a step (d) of measuring the in-situ magnetic property of said metal specimen, whereby corresponding to the temperature variation, both the expansion curve and the magnetic property change of said metal specimen can be obtained.

8. A method as claimed in Claim 3 wherein said heated metal specimen is cooled, within a short time, to a temperature where a phase transformation of said specimen may be caused, and then kept in an isothermal condition for a pre-determined time.

9. A measuring apparatus comprising:

means for mounting a metal specimen having a vacuum housing thereon so that said metal specimen can be measured under the vacuum condition;

a programmable controlling means for pre-setting the time-temperature patterns of the measurement so as to prescribe a desired heat treatment;

means controlled by said programmable controlling means for triggering said metal specimen and then measuring the internal friction behavior of said metal specimen;

means controlled by said programmable controlling means for controlling the temperature variation in said vacuum housing; and

means for recording the measured data of the temperature and of the internal friction seperately from said temperature controlling means and said

triggering and measuring means; whereby the in-situ internal friction behavior of said metal specimen can be obtained during the phase transformation of said metal specimen.

10. An apparatus as claimed in Claim 9 wherein said temperature measuring means includes a heating device and a cooling device.

11. An apparatus as claimed in Claim 9 wherein said triggering and measuring means is an inverse torsion pendulum internal friction testing device.

12. An apparatus as claimed in Claim 9 wherein said temperature controlling means further comprises a temperature sensor whose one end is provided in the torsion center of said metal specimen so as to avoid causing any unexpected external friction.

13. An apparatus as claimed in Claim 9 further comprising a dilatometer for measuring the thermal expansion of said metal specimen at different temperatures.

14. An apparatus as claimed in Claim 13 wherein said dilatometer includes a solenoid coil and a core which is inserted into said coil.

15. An apparatus as claimed in Claim 9 further comprising a means for measuring the magnetic property of said metal specimen at different temperatures.

16. An apparatus as claimed in Claim 15 wherein said magnetic measuring means includes an induction coil

surrounded around said metal specimen.

17. An apparatus as substantially described hereinbefore with reference to the accompanying drawings.

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